METHOD 8270C/8015M (DRO)

1.0 SCOPE AND APPLICATION

1.1 This method can be used to quantitate most basic, neutral and acidic organic compounds (BNAs) that are soluble in methylene chloride and capable of being eluted, without derivatization, as sharp peaks from a gas chromatographic fused-silica capillary column coated with a slightly polar silicone. Such compounds include polynuclear aromatic hydrocarbons (PNAs), chlorinated hydrocarbons and pesticides, phthalate esters, organophosphate esters, nitrosamines, haloethers, aldehydes, ethers, ketones, anilines, pyridines, quinolines, aromatic nitro compounds, and phenols, including nitrophenols. See Tables 1a and 1b for a list of compounds (along with their characteristic ions) that have been evaluated.

1.2 In most cases, this method is not appropriate for the quantitation of multicomponent analytes, e.g., Aroclors, Toxaphene, Chlordane, *etc*., because of limited sensitivity for those analytes. When these analytes have been identified by another technique, this method is appropriate for confirmation of the presence of these analytes when concentration in the extract permits. However, this method is used for the analysis of petroleum hydrocarbons, namely diesel range organics (DROs). DROs correspond to the range of alkanes from C_{10} to C_{28} and covering a boiling point range of approximately 170°C - 430°C. The identification of specific fuel types may be complicated by environmental processes such as evaporation, biodegradation, or when more than one fuel type is present.

1.3 The following compounds may require special treatment when being determined by this method:

- Benzidine may be subject to oxidative losses during solvent concentration and its chromatographic behavior is poor.
- Under the alkaline conditions of the extraction step from aqueous matrices, α-BHC, β-BHC, Endosulfan I and II, and Endrin are subject to decomposition. Neutral extraction should be performed if these compounds are expected.
- Hexachlorocyclopentadiene is subject to thermal decomposition in the inlet of the gas chromatograph, chemical reaction in acetone solution, and photochemical decomposition.
- N-nitrosodimethylamine is difficult to separate from the solvent under the chromatographic conditions described.
- N-nitrosodiphenylamine decomposes in the gas chromatographic inlet and cannot be separated from diphenylamine.
- Pentachlorophenol, 2,4-dinitrophenol, 4-nitrophenol, benzoic acid, 4,6-dinitro-2-methylphenol, 4-chloro-3-methylphenol, 2-nitroaniline, 3-nitroaniline, 4-chloroaniline, and benzyl alcohol are subject to erratic chromatographic behavior, especially if the GC system is contaminated with high boiling material.
- Pyridine may perform poorly at the GC injection port temperatures listed in the method. Lowering the injection port temperature may reduce the amount of degradation. The analyst needs to use caution if modifying the injection port temperature as the performance of other analytes may be adversely affected.
- In addition, analytes in the list provided above are flagged when there are limitations caused by sample preparation and/or chromatographic problems.

1.4 The estimated quantitation limit (PQL) of Method 8270/8015 for determining an individual compound is approximately 330 µg/kg (wet weight) for soil/sediment samples, 1-200 mg/kg for wastes (dependent on matrix and method of preparation), and 10 µg/L for ground water samples (see Tables 2a and 2b). For DROs, the values are 4 mg/kg (soil/sediment, wet weight) and 0.1 mg/L for ground water (see Table 2c). PQLs will be proportionately higher for sample extracts that require dilution to avoid saturation of the detector.

1.5 This method is restricted to use by or under the supervision of analysts experienced in the use of gas chromatograph/mass spectrometers and skilled in the interpretation of mass spectra. Each analyst must demonstrate the ability to generate acceptable results with this method.

2.0 SUMMARY OF METHOD

2.1 The samples are prepared for analysis by gas chromatography/mass spectrometry (GC/MS) using the appropriate sample preparation (refer to Methods 3500, 3510, 3550).

2.2 The semivolatile compounds are introduced into the GC/MS by injecting the sample extract into a gas chromatograph (GC) with a narrow-bore fused-silica capillary column. The GC column is temperature-programmed to separate the analytes, which are then detected with a mass spectrometer (MS) connected to the gas chromatograph.

2.3 Analytes eluted from the capillary column are introduced into the mass spectrometer via a jet separator or a direct connection. Identification of target analytes is accomplished by comparing their mass spectra with the electron impact spectra of authentic standards. Quantitation is accomplished by comparing the response of a major (quantitation) ion relative to an internal standard using a five-point (or more) calibration curve.

2.4 The method includes specific calibration and quality control steps that supersede the general requirements provided in Method 8000.

3.0 INTERFERENCES

3.1 Raw GC/MS data from all blanks, samples, and spikes must be evaluated for interferences. Determine if the source of interference is in the preparation and/or cleanup of the samples and take corrective action to eliminate the problem.

3.2 Contamination by carryover can occur whenever high-concentration and low-concentration samples are sequentially analyzed. This is virtually never a problem for direct injection GC/MS analysis, provided that the sample syringe is rinsed with solvent between sample injections. If carryover is suspected, questionable samples should be re-injected after the system has been demonstrated to be free of contamination.

4.0 APPARATUS AND MATERIALS

4.1 Gas chromatograph/mass spectrometer system

4.1.1 Gas chromatograph - An analytical system complete with a temperature-programmable gas chromatograph suitable for splitless injection and all required accessories, including syringes, analytical columns, and gases. The capillary column should be directly coupled to the source.

4.1.2 Column - 30 m × 0.25 mm ID × 0.5 µm film thickness silicone-coated fused-silica capillary column (Hewlett Packard HP-5ms SV or equivalent).

4.1.3 Mass spectrometer capable of scanning from 35 to 500 amu every 1 sec or less, using 70 volts (nominal) electron energy in the electron impact ionization mode. The mass spectrometer must be capable of producing a mass spectrum for decafluorotriphenylphosphine (DFTPP) which meets the criteria in Table 4 when 1 µL of the GC/MS tuning standard is injected through the GC (50 ng of DFTPP)

4.1.4 GC/MS interface - Any GC-to-MS interface may be used that gives acceptable calibration points at 50 ng per injection for each compound of interest and achieves acceptable tuning performance criteria. For a narrow-bore capillary column, the interface is usually capillary-direct into the mass spectrometer source.

4.1.5 Data system - A computer system is interfaced to the mass spectrometer. Hewlett-Packard Chemstation software (with environmental data analysis) is used to acquire and process GC/MS data.

4.2 Syringes - 1 to 1000 µL Hamilton syringes are used in the preparation of standards, spiking solutions, and standards.

4.3 Balance - Analytical, capable of weighing 0.0001 g.

4.4 Bottles - glass with polytetrafluoroethylene (PTFE)-lined screw caps or crimp tops.

5.0 REAGENTS

5.1 Reagent grade inorganic chemicals shall be used in all tests. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without introducing adverse interferences.

5.2 Organic-free reagent water - All references to water in this method refer to organic-free reagent water.

5.3 Stock standard solutions

5.3.1 Certified stock standard solutions are purchased when available for the bulk of desired analytes. They are typically available at concentrations of 1000 to 2000 mg/L.

5.3.2 Supplemental compounds added to calibration mixes are generally prepared gravimetrically from neat standard references (in order to create a stock solution of 1000 to 10000 mg/L).

5.3.3 Stock standard solutions are stored in bottles with PTFE-lined screw-caps. They are refrigerated and protected from light, as recommended by the standard manufacturer.

5.3.4 Neat standard references are used in order to create a stock solution ~10000 mg/L for DRO standards.

5.3.5 Stock standard solutions are replaced prior to expiration, or sooner if comparison with quality control check samples indicates a problem.

5.4 Internal standard solutions - The internal standards used are 1,4-Dichlorobenzene-d_4, Naphthalene-d_8, Acenaphthene-d_{10}, Phenanthrene-d_{10}, Chrysene-d_{12}, and Perylene-d_{12}. Internal standards are spiked into the sample extracts and calibration standards at a uniform concentration of 40 ng/µL (≡ 40 mg/L).

5.5 GC/MS tuning standard - A methylene chloride solution containing 50 ng/µL of decafluorotriphenylphosphine (DFTPP), pentachlorophenol, and benzidine is used to evaluate GC/MS tuning criteria, injection port inertness, and GC column performance.

5.6 Calibration standards - A minimum of five calibration standards should be prepared at five different concentrations. If possible, the lowest calibration standard corresponds to a sample concentration at or below the standard reporting limit. The remaining standards should correspond to the working range of the GC/MS system (10-100 ng/µL for BNAs, 2-100 ng/µL for PNAs, and 0.1-1.0 g/L for DROs). Each standard should contain each analyte for detection by this method. The preparation instructions for the creation of calibrations standards from stock solutions commonly used in BNA, PNA, and DRO analysis are found in the Calibration Recipes excel file.

5.7 Surrogate standards - The surrogates used are Phenol-d_6, 2-Fluorophenol, 2,4,6-Tribromophenol, Nitrobenzene-d_5, 2-Fluorobiphenyl, and p-Terphenyl-d_{14}. See Method 3500 for instructions on preparing the surrogate solutions. For BNA analysis, all six surrogate standards are used. For PNA analysis, only Nitrobenzene-d_5, 2-Fluorobiphenyl, and p-Terphenyl-d_{14} are used.

5.7.1 Surrogate spiking mixes are created at 100 mg/L and spiked into samples in 1 mL aliquots. The resulting ideal concentration in a 1 mL extract as 100ng/µL.

5.7.2 Surrogate Standard Check: inject a sample of the spiking solution (with internal standard added) into the GC/MS to determine recovery of surrogate standards. It is recommended that this check be done whenever a new surrogate spiking solution is prepared.

5.8 Matrix spike and laboratory control standards - See Method 3500 for instructions on preparing the matrix spike standard. The same standard is used as the laboratory control standard (LCS).

5.8.1 Matrix spiking mixes for single component analytes are generally created at 50 mg/L and spiked into samples in 1 mL aliquots. The resulting ideal concentration in a 1 mL extract is 50ng/µL.

5.8.2 The matrix spiking mix for DRO is created at 1000 mg/L and spiked into samples in 1 mL aliquots. The resulting ideal concentration in a 1 mL extract is 1000 ng/µL.

5.8.3 Matrix Spike Check: inject a sample of the spiking solution (with internal standard added) into the GC/MS to determine recovery of surrogate standards. It is recommended that this check be done whenever a new surrogate spiking solution is prepared.

5.9 Acetone, hexane, methylene chloride, isooctane, carbon disulfide, toluene, and other appropriate solvents - All solvents should be pesticide quality or equivalent.

6.0 SAMPLE COLLECTION, PRESERVATION, AND HANDLING

6.1 Unanalyzed sample extracts are refrigerated and protected from light in sealed vials.

6.2 The holding time for samples is 7 days until extraction and 40 days thereafter.

6.3 Samples and target compound standards are stored separately.

7.0 PROCEDURE

7.1 Sample preparation

7.1.1 Soil and water are normally prepared via an extraction procedure (*cf.* Methods 3500, 3510, 3550; SOPs: 3510bna.doc and 3550bna.doc) prior to GC/MS analysis.

7.1.2 In cases where the sample is an oil or a solvent, the sample is simply diluted with methylene chloride and analyzed at a concentration appropriate for the level of analytes/interferences in the sample. In this situation, the sample is reported on a weight basis and analyzed in a batch of soil samples.

7.2 GC/MS operating conditions - see Table 3 for routine operating conditions for both BNA and PNA analysis.

7.3 Initial calibration

7.3.1 The GC/MS system must be hardware-tuned using a 50 ng injection of DFTPP prior to the analysis of calibration standards and samples.

7.3.1.1 In the absence of any other manipulations, evaluate the mass spectrum of the highest intensity level from the total ion chromatogram for the DFTPP peak. This is the default approach used.

7.3.1.2 If the above evaluation is adversely affected by ion peak asymmetry, average the three highest intensity scans of the peak or average the mass spectrum ranging from the 10% initial peak intensity to the tailing 10% peak intensity level from the total ion chromatogram for the DFTPP peak.

7.3.1.3 If the above evaluation is adversely affected by background contamination, perform a background subtraction with a spectrum within 20 scans of the DFTPP peak which does not represent a target compound. Use of this procedure may be indicative of failing MS performance. The MS source should be cleaned and re-tuned.

7.3.1.4 The DFTPP mass intensity criteria in Table 4 are used as tuning acceptance criteria.

7.3.1.5 All subsequent standards, samples, MS/MSDs, and blanks associated with a DFTPP analysis must use the identical mass spectrometer instrument conditions.

7.3.1.6 The GC/MS tuning standard solution should also be used to assess GC column performance and injection port inertness. Benzidine and pentachlorophenol should be present at their normal responses, and peak tailing should be minimal.

7.3.1.7 The injection port is replaced prior to any calibration sequence or 12-hour BNA analytical sequence. If chromatography is still poor, it may also be necessary to clip off the first 6-12 in. of the capillary column. Clipping the column may necessitate recalibration, as retention times and responses can shift considerably.

7.3.2 Analyze 1-2 µL of each calibration standard (containing internal standards) and tabulate the area of the primary characteristic ion against concentration for each target analyte (as indicated in Tables 1a and 1b). A set of at least five calibration standards is necessary. The injection volume must be the same for all standards and sample extracts. Figure 1 shows a chromatogram of a calibration standard containing base/neutral and acid analytes. Depending on the current state of the GC/MS system, a 2 µL injection may be required to achieve appropriate responses for the lower levels of the least sensitive compounds. When possible, however, a 1 µL can be used to yield a better dynamic range for the high sensitivity compounds.

7.3.3 Calculate response factors (*RF*s) for each target analyte relative to one of the internal standards as follows: $RF = A_s C_i / A_i C_s$. Here, A_s and A_i are the areas of the standard compound and corresponding internal standard, respectively. Likewise, C_s and C_i are the respective concentrations (in any consistent set of units) of the standard compound and corresponding internal standard.

7.3.4 System performance check compounds (SPCCs)

7.3.4.1 A system performance check must be performed to ensure that minimum *RF*s are met before the calibration curve is used. For semivolatiles, the System Performance Check Compounds (SPCCs) are: N-nitroso-di-n-propylamine; hexachlorocyclopentadiene; 2,4-dinitrophenol; and 4-nitrophenol.

7.3.4.2 The minimum acceptable average *RF* for these compounds is 0.050. These SPCCs typically have low *RF*s (0.1-0.2) and tend to decrease in response as the chromatographic system begins to deteriorate or the standard material begins to deteriorate. They are usually the first to show poor performance. Therefore, they must meet the minimum requirement when the system is calibrated.

7.3.4.3 If the minimum response factors are not met, the system must be evaluated, and corrective action must be taken before sample analysis begins. Possible problems include standard mixture degradation, contamination at the front end of the analytical column, and active sites in the column or chromatographic system. Replacing the calibration standards, and/or clipping/replacing the column will likely solve this problem.

7.3.5 Calibration check compounds (CCCs)

7.3.5.1 The purpose of the CCCs is to evaluate the calibration from the standpoint of the integrity of the system. High variability for these compounds may be indicative of system leaks or reactive sites on the column. Meeting the CCC criteria is in addition to the successful calibration of the target analytes using one of the approaches described in Section 7.0 of Method 8000.

7.3.5.2 Calculate the mean response factor and the relative standard deviation (RSD) of the response factors for each target analyte. The RSD should be less than or equal to 15%

for each target analyte. However, the RSD for each individual CCC (see Semi Volatile QC Reference) must be less than or equal to 30%.

7.3.5.3 If the RSD of any CCC is greater than 30%, then the chromatographic system is too reactive for analysis to begin. Clean or replace the injector liner and/or capillary column, then repeat the calibration procedure.

7.3.6 Evaluation of retention times - The relative retention time (RRT) of each target analyte in each calibration standard should agree within 0.06 RRT units. This is accomplished by setting the retention time extraction windows in the Chemstation software.

7.3.7 Linearity of target analytes - If the %RSD of any target analytes is 15% or less, then the relative response factor may be assumed to be constant over the calibration range, and the average relative response factor may be used for quantitation

7.3.7.1 Refer to Method 8000 if a least-squares regression is used to determine a linear or quadratic fit to the calibration data. Note that quadratic polynomials are generally fit through the origin in order to prevent the symptomatic aphysical prediction of high concentrations at very low responses. (All least-squares regressions used for OHIO VAP analysis will use a calculated intercept and quadratic fits will only be used for compounds exhibiting nonlinear behavior). In any event, the COD for any regression fit should be ≥0.99. In addition, 6 calibration data points are required for a calibration fit with 3 free parameters, while 5 are required for a calibration fit with 1 or 2 free parameters.

7.3.7.2 When the RSD exceeds 15%, the plotting and visual inspection of a calibration curve can be a useful diagnostic tool. The inspection may indicate analytical problems, including errors in standard preparation, the presence of active sites in the chromatographic system, analytes that exhibit poor chromatographic behavior, *etc*.

7.3.7.3 The quality of the calibration fit for any particular compound is communicated to the data user via the Quality Control report for a given batch of samples. The calibration summary report includes: the concentration and RF for each standard in the calibration curve, the type of calibration fit, the calibration fit parameters (*i.e*. average RF or regression coefficients), and the appropriate calibration quality metric (*i.e*. %RSD or COD).

7.4 GC/MS calibration verification - Calibration verification consists of three steps that are performed at the beginning of each 12-hour analytical shift.

7.4.1 Prior to the analysis of samples or calibration standards, inject 50 ng of the DFTPP standard into the GC/MS system. The resultant mass spectrum for DFTPP must meet the criteria given in Table 4 before sample analysis begins. These must be *injected* within 12 hours of the injection time for the DFTPP.

7.4.2 The initial calibration for each compound of interest should be verified once every 12 hours prior to sample analysis, using the introduction technique and conditions used for samples. This is accomplished by analyzing a calibration standard at a concentration near the midpoint concentration (50 ng/µL) for the calibration range of the GC/MS. The results from the calibration standard analysis must meet the verification acceptance criteria provided below for the SPCC and CCC compounds.

7.4.3 A method blank is run every 20 samples to ensure that the total system (preparation glassware, introduction device, transfer lines, and the GC/MS system itself) is free of contaminants.

7.4.4 System performance check compounds (SPCCs)

7.4.4.1 A system performance check must be made during every 12-hour analytical shift. Each SPCC in the calibration verification standard must meet a minimum response factor of 0.050. This is the same check that is applied during the initial calibration.

7.4.4.2 If the minimum response factors are not met, the system must be evaluated, and corrective action must be taken before sample analysis begins (see previous).

7.4.5 Calibration check compounds (CCCs)

7.4.5.1 After the system performance check is met, the CCCs listed in Semi Volatile Organic QC Reference are used to check the validity of the initial calibration. Percent drift is used to evaluate the CCC response and it must be ≤20%. Drift is defined as the normalized deviation of the measured from the spike value of a target component: $\%D = \left|C - C_{spike}\right| / C_{spike}$.

7.4.5.2 If the percent drift for each CCC is ≤20%, then the initial calibration is assumed to be valid. If the criterion is not met for any one CCC, then corrective action must be taken prior to the analysis of samples (see previous).

7.4.5.3 Problems similar to those listed under SPCCs could affect the CCCs. If the problem cannot be corrected by other measures, a new initial calibration must be generated. The CCC criteria must be met before sample analysis begins.

7.5 GC/MS analysis of samples

7.5.1 Samples are screened at a diluted state via GC/MS whenever possible prior to analysis within a 12-hour QC batch. This can identify potentially low surrogate recoveries, high target compound concentrations, non-target matrix interferences. This will minimize contamination of the GC/MS system from unexpectedly high concentrations of organic compounds.

7.5.2 Allow the sample extract to warm to room temperature. 3 µL of the internal standard solution is added to a 300 µL aliquot of sample in a crimp-top vial for subsequent autosampler analysis. If the sample extract is to be diluted prior to analysis, a smaller sample volume is used (even though the net volume of nominally 300 µL remains the same). The Dilution Reference has a list of commonly used dilutions as well as the required amount of sample, solvent, and internal standard for each dilution.

7.5.3 Inject a 1-2 µL aliquot of the sample extract into the GC/MS system, using the same operating conditions that were used for the calibration.

7.5.4 If the response for any quantitation ion exceeds the initial calibration range of the GC/MS system by more than 10%, the sample extract should be diluted and reanalyzed. In any event, a result based on an extrapolation of calibration curve beyond the working range is flagged on the analytical report.

7.5.5 The EICP area for all of the internal standards in all spikes, blanks, and samples is monitored relative to the most recent calibration verification standard. Changes by more than a factor of two (*i.e.* 50% to 200%) can indicate adverse matrix effects (in the case of an isolated sample) or degrading MS performance (in the case of a systematic low bias). A single-sample matrix effect is documented either via screening or re-analysis and is noted on the analytical report (see Semi Volatile QC Reference). Similarly, the retention times for all of the internal standards in all spikes, blanks, and samples is monitored relative to the most recent calibration verification standard. The change in retention time for any internal standard by more than 30 seconds of the most recent calibration verification standard is indicative of the same potential problems listed above and should be flagged/corrected as appropriate.

7.6 Qualitative analysis

7.6.1 The qualitative identification of compounds determined by this method is based on retention time and on comparison of the sample mass spectrum, after background correction, with characteristic ions in a reference mass spectrum. The reference mass spectrum must be generated by the laboratory using the conditions of this method. The characteristic ions from the reference mass spectrum are given in Tables 1a and 1b. Compounds are identified when the following criteria are met.

7.6.1.1 Initial selection of a target compound peak is performed by the Chemstation data system search routine. The search is based on the presence of a target chromatographic peak containing ions specific for the target compound at a compound-specific retention time.

7.6.1.2 The RRT of the sample component is within ± 0.06 RRT units of the RRT of the standard component. This is accomplished using retention time extraction windows within the Chemstation data system.

7.6.1.3 The relative intensities of the characteristic ions agree within 30% of the relative intensities of these ions in the reference spectrum. (Example: For an ion with an abundance of 50% in the reference spectrum, the corresponding abundance in a sample spectrum can range between 20% and 80%.)

7.6.1.4 Structural isomers that produce very similar mass spectra are identified as individual isomers if they have sufficiently different GC retention times. Sufficient GC resolution is achieved if the height of the valley between two isomer peaks is less than 25% of the sum of the two peak heights. Otherwise, structural isomers are identified as isomeric pairs.

7.6.1.5 Identification is hampered when sample components are not resolved chromatographically and produce mass spectra containing ions contributed by more than one analyte. When gas chromatographic peaks obviously represent more than one sample component (*i.e.*, a broadened peak with shoulder(s) or a valley between two or more maxima), appropriate selection of analyte spectra and background spectra is important.

7.6.1.6 Examination of extracted ion current profiles of appropriate ions can aid in the selection of spectra and in qualitative identification of compounds. When analytes coelute (*i.e.*, only one chromatographic peak is apparent), the identification criteria may be met, but each analyte spectrum will contain extraneous ions contributed by the coeluting compound.

7.6.1.7 In the two previous cases, analyst expertise as well as knowledge of site history may be important in accepting/rejecting the identification of a compound. In the event of continued uncertainty, the analyst should preferentially make a conservative judgement and accept an identified hit, allowing the potential for a false positive.

7.6.2 For samples containing components not associated with the calibration standards, a library search may be made for the purpose of tentative identification. The necessity to perform this type of identification will be determined by the purpose of the analyses being conducted. Only after visual comparison of sample spectra with the nearest library searches may the analyst assign a tentative identification. Guidelines for tentative identification are:

- Relative intensities of major ions in the reference spectrum (ions > 10% of the most abundant ion) should be present in the sample spectrum.
- The relative intensities of the major ions should agree within ± 20%.
- Molecular ions present in the reference spectrum should be present in the sample spectrum.

- Ions present in the sample spectrum but not in the reference spectrum should be reviewed for possible background contamination or presence of coeluting compounds.
- Ions present in the reference spectrum but not in the sample spectrum should be reviewed for possible subtraction from the sample spectrum because of background contamination or coeluting peaks. Data system library reduction programs can sometimes create these discrepancies.

7.7 Quantitative analysis

7.7.1 Once a compound has been identified, the quantitation of that compound will be based on the integrated abundance of the primary characteristic ion from the EICP.

7.7.2 The curve fit applied in the initial calibration is the same as that used to compute the concentration of a target analyte in a sample. All curve fits are evaluated by the data system and are of the form: $A_s/A_i = k_0 + k_1[C_s/C_i] + k_2[C_s/C_i]^2$. Here A_s and A_i are the areas of the target and internal standard, C_s and C_i are the concentrations of the target and internal standard, and k_i is the i^{th}-order regression coefficient. Note that for a mean RF fit to the calibration data, $k_1 \equiv$ <RF>, while k_0, $k_2 \equiv 0$.

7.7.3 The concentration of any non-target analytes identified in the sample may be estimated by assuming a mean RF of 1 and by using the TIC areas for the nearest internal standard and target compound. The resulting concentration should be reported indicating: (1) that the value is an estimate, and (2) which internal standard was used to determine concentration. Use the nearest internal standard free of interferences.

7.7.4 Structural isomers that produce very similar mass spectra should be quantitated as individual isomers if they have sufficiently different GC retention times. Otherwise, structural isomers are quantitated as isomeric pairs (such as p- and m-cresol).

7.8 Special procedures for diesel range organics (DROs) - The following items detail the differences in the calibration procedures for multicomponent, diesel range organics from the general, single component procedures outlined above.

7.8.1 A set of at least five calibration standards per §7.2 and §7.3.2. The preparation instructions for the creation of the DRO calibration standards can be found in the Calibration Recipes excel file. Figure 2 represents a DRO chromatogram.

7.8.2 Instead of using the measured responses from the TIC to represent the DRO calibration, the total area of ions characteristic to the fuel in question is used in an internal standard calibration. Using characteristic ions to represent a fuel has the advantage of reducing biases from other components present in the chromatogram (namely surrogate and internal standards), and the use of an internal standard improves the stability of a calibration. Diesel range organics consist primarily of straight and branched alkanes (~C_{10} to C_{28}). For the alkanes, the characteristic mass used is m/z = 57 (corresponding to the ion $C_4H_9^+$). This single ion is used for quantitation.

7.8.3 Calculate response factors (*RF*s) for each target mass relative to one of the internal standards as follows: $RF = A_s C_i / A_i C_s$. Here, A_s and A_i are the areas of the characteristic mass over its respective time range and corresponding internal standard, respectively. Likewise, C_s and C_i are the respective concentrations (in any consistent set of units) of the *total fuel* and corresponding internal standard. The characteristic time ranges for each mass depend upon current chromatographic conditions. The time ranges used for the sample chromatograms in Figures 1 and 2 are listed in the respective figure.

7.8.4 The relative area response vs. relative concentration for each characteristic mass is calibrated in the same manner as described in §7.3.7 and §7.7.

7.8.5 The net result for a DRO analysis is based upon the single characteristic mass (57).

7.8.6 In addition the routine SPCC/CCC check standard, a DRO check standard is analyzed in order characterize the efficacy of the present DRO calibration in terms of recovery and retention times.

8.0 QUALITY CONTROL

8.1 All of the quality control items employed and evaluated are listed in Semi Volatile QC Reference. In addition, the Semi Volatile QC Reference indicates the frequency of each QC item along with appropriate courses of corrective action.

8.2 Quality control items are inspected by the analyst as the data becomes available. At the conclusion of the analytical batch, all of the samples, spikes, standards, *etc*. are processed and evaluated automatically and stored electronically for future reference/retrieval.

9.0 METHOD PERFORMANCE

9.1 Laboratory-specific performance data is provided in this document

9.2 Tables 2a, 2b, and 2c present the results for the most recent detection limit studies. The MDL, PQL and ratio of PQL/MDL are given for each analyte in the BNA and PNA target compound lists.

9.3 Lower and upper acceptance limits for all surrogate and matrix spiking compounds can be found in the Semi Volatile QC Reference.

10.0 REFERENCES

1. SW-846, 1996, Revision 3; Methods 3500, 3510, 3550, 8000, 8015, 8270.

11.0 APPROVAL AND ISSUE

Analyst	Date
Andy Ball, QA Officer	Date
Maya V. Murshak, Technical Director	Date

12.0 TABLES AND FIGURES

Table 1a. Summary of Retention Times† and Characteristic Ions for Base/Neutral/Acid Semi-Volatile Organics

#	**Compound**	t_R (*min*)	$t_R/t_{R,I}$ (-)	$t_R-t_{R,I}$ (*min*)	1^0 *m/z*	2^0 *m/z*	3^0 *m/z*	4^0 *m/z*
1)	1,4-DICHLOROBENZENE-D4	6.92	1.000	0.00	152	150	115	
2)	N-Nitrosodimethylamine	3.79	0.548	-3.13	74	42	43	
3)	2-Picoline	4.61	0.666	-2.31	93	92	66	
4)	Methyl methanesulfonate	5.16	0.746	-1.76	80	79	95	
5)	2-Fluorophenol	5.31	0.767	-1.61	112	92	64	
6)	Ethyl methanesulfonate	5.99	0.866	-0.93	79	109	97	
7)	Phenol-d5	6.45	0.932	-0.47	99	71	42	
8)	Phenol **	6.47	0.935	-0.45	94	66	65	
9)	Aniline	6.53	0.944	-0.39	93	65	66	
10)	2-Chlorophenol **	6.68	0.965	-0.24	128	130	92	
11)	Bis(2-chloroethyl)ether	6.59	0.952	-0.33	93	63	95	
12)	1,3-Dichlorobenzene	6.94	1.003	0.02	146	148	111	
13)	1,4-Dichlorobenzene **	6.94	1.003	0.02	146	148	111	
14)	1,2-Dichlorobenzene	7.18	1.038	0.26	146	148	111	
15)	Bis(2-chloroisopropyl)ethe	7.3	1.055	0.38	45	121	77	
16)	Benzyl Alcohol	7.09	1.025	0.17	108	107	79	77
17)	Acetophenone	7.46	1.078	0.54	105	77	120	
18)	o-Cresol	7.24	1.046	0.32	108	107	79	
19)	p,m-Cresol	7.42	1.072	0.50	107	108	79	
20)	Hexachloroethane	7.59	1.097	0.67	117	119	201	
21)	N-Nitrosodi-n-propylamine*	7.48	1.081	0.56	70	43	42	41
22)	NAPHTHALENE-D8	8.59	1.000	0.00	136	68	108	
23)	Nitrobenzene-d5	7.66	0.892	-0.93	82	128	98	
24)	Nitrobenzene	7.69	0.895	-0.90	123	123	65	
25)	N-Nitrosopiperidine	7.89	0.919	-0.70	114	55	56	
26)	Isophorone	7.98	0.929	-0.61	82	95	138	
27)	2-Nitrophenol	8.11	0.944	-0.48	139	109	65	
28)	2,4-Dimethylphenol	8.12	0.945	-0.47	107	122	121	
29)	Bis(2-chloroethoxy)methane	8.25	0.960	-0.34	93	63	95	
30)	2,4-Dichlorophenol	8.4	0.978	-0.19	162	164	98	
31)	1,2,4-Trichlorobenzene **	8.52	0.992	-0.07	180	182	145	
32)	Naphthalene	8.61	1.002	0.02	128	127	129	
33)	4-Chloroaniline	8.69	1.012	0.10	127	129	65	
34)	2,6-Dichlorophenol	8.71	1.014	0.12	162	164	127	
35)	Hexachlorobutadiene	8.83	1.028	0.24	225	223	227	
36)	N-Nitroso-di-n-butylamine	9.13	1.063	0.54	84	116	158	
37)	4-Chloro-3-methylphenol **	9.28	1.080	0.69	107	142	77	
38)	2-Methylnaphthalene	9.5	1.106	0.91	141	142	115	
40)	1,2,4,5-Tetrachlorobenzene	9.77	1.137	1.18	216	214	218	
41)	Hexachlorocyclopentadiene	9.79	1.140	1.20	237	235	239	
42)	2,4,6-Trichlorophenol	9.94	1.157	1.35	196	198	200	
43)	2,4,5-Trichlorophenol	9.94	1.157	1.35	196	198	132	
44)	2-Fluorobiphenyl	9.98	1.162	1.39	172	171	173	
45)	2-Chloronaphthalene	10.13	1.179	1.54	162	127	164	
46)	1-Chloronaphthalene	10.18	1.185	1.59	162	164	127	
47)	2-Nitroaniline	10.29	1.198	1.70	138	65	92	

#	**Compound**	t_R (*min*)	$t_R/t_{R,I}$ (-)	t_R-$t_{R,I}$ (*min*)	1^0 *m/z*	2^0 *m/z*	3^0 *m/z*	4^0 *m/z*
48)	Acenaphthylene	10.67	1.242	2.08	152	151	150	
49)	Dimethyl Phthalate	10.54	1.227	1.95	163	164	77	
50)	2,6-Dinitrotoluene	10.64	1.239	2.05	165	121	148	
51)	3-Nitroaniline	10.82	1.260	2.23	138	92	65	
52)	Acenaphthene **	10.91	1.270	2.32	154	153	152	
53)	2,4-Dinitrophenol	10.95	1.275	2.36	184	63	107	
54)	Dibenzofuran	11.12	1.295	2.53	168	169	139	
55)	4-Nitrophenol **	10.99	1.279	2.40	65	139	81	109
56)	2,4-Dinitrotoluene **	11.15	1.298	2.56	165	89	63	
57)	2,3,4,6-Tetrachlorophenol	11.32	1.318	2.73	232	230	234	131
58)	Fluorene	11.56	1.346	2.97	166	165	167	
59)	Diethyl phthalate	11.43	1.331	2.84	149	150	177	
60)	4-Chlorophenyl phenyl ethe	11.53	1.342	2.94	204	141	206	
61)	4-Nitroaniline	11.61	1.352	3.02	138	65	92	
62)	2,4,6-Tribromophenol	11.89	1.384	3.30	330	332	141	
63)	PHENANTHRENE-D10	12.8	1.000	0.00	188	80	94	
64)	4,6-Dinitro-2-methylphenol	11.67	0.912	-1.13	198	121	105	
65)	N-Nitrosodiphenylamine	11.69	0.913	-1.11	169	168	167	
66)	1,2-Diphenylhydrazine	11.74	0.917	-1.06	182	105	77	
67)	Azobenzene	11.74	0.917	-1.06	182	105	77	
68)	4-Bromophenyl phenyl ether	12.16	0.950	-0.64	248	250	77	
69)	1,3,5-Trinitrobenzene	12.08	0.944	-0.72	213	74	75	
70)	Phenacetin	12.11	0.946	-0.69	108	109	179	137
71)	Hexachlorobenzene	12.37	0.966	-0.43	249	282	286	
72)	4-Aminobiphenyl	12.51	0.977	-0.29	169	168	170	
73)	Pentachlorophenol **	12.6	0.984	-0.20	266	264	268	
75)	Pronamide	12.6	0.984	-0.20	173	174	145	
76)	Pentachloronitrobenzene	12.72	0.994	-0.08	237	249	295	
77)	Phenanthrene	12.83	1.002	0.03	178	176	179	
78)	Anthracene	12.83	1.002	0.03	178	176	179	
79)	di-N-butyl phthalate	13.62	1.064	0.82	149	150	104	
80)	Fluoranthene	14.65	1.145	1.85	202	200	203	
81)	Terphenyl-d14	15.28	1.194	2.48	244	122	212	
82)	CHRYSENE-D12	17.34	1.000	0.00	240	236	120	
83)	Benzidine	14.83	0.855	-2.51	184	185	92	
84)	Pyrene **	15.03	0.867	-2.31	202	200	203	
85)	p-Dimethylaminoazobenzene	15.57	0.898	-1.77	225	120	77	
86)	Butyl benzyl phthalate	16.23	0.936	-1.11	149	91	206	
88)	Benzo(a)anthracene	17.29	0.997	-0.05	228	226	229	
89)	3,3'-Dichlorobenzidine	17.25	0.995	-0.09	252	254	126	
90)	Chrysene	17.38	1.002	0.04	228	226	229	
92)	PERYLENE-D12	20.3	1.000	0.00	264	260	265	
91)	Bis(2-ethylhexyl)phthalate	17.42	0.858	-2.88	149	167	279	
93)	Di-n-octyl phthalate	18.66	0.919	-1.64	149	43	167	
95)	Benzo(b)fluoranthene	19.47	0.959	-0.83	252	253	126	
96)	7,12-Dimethylbenz(a)anthra	19.5	0.961	-0.80	256	241	239	
97)	Benzo(k)fluoranthene	19.47	0.959	-0.83	252	253	126	

#	**Compound**	t_R (*min*)	$t_R/t_{R,I}$ (-)	t_R-$t_{R,I}$ (*min*)	1^0 *m/z*	2^0 *m/z*	3^0 *m/z*	4^0 *m/z*
98)	Benzo(a)pyrene	20.16	0.993	-0.14	252	253	126	
99)	3-Methylcholanthrene	21.04	1.036	0.74	268	252	253	269
100)	Dibenz(a,j)acridine	22.54	1.110	2.24	279	280	139	278
101)	Indeno(1,2,3-cd)pyrene	23.1	1.138	2.80	276	138	277	
102)	Dibenzo(a,h)anthracene	23.14	1.140	2.84	278	139	279	
103)	Benzo(ghi)perylene	23.92	1.178	3.62	276	138	277	

†: Absolute retention times (t_R) listed are from calibration BU061002.M. Absolute and relative retention times ($t_R/t_{R,I}$) may shift with the present condition of the column (*i.e*. new, clipped, *etc*.), but the differential retention times (t_R - $t_{R,I}$) tend to remain constant given the same chromatographic temperature program.

Table 1b. Summary of Retention Times† and Characteristic Ions for Polynuclear Aromatic Semi-Volatile Organics

#	**Compound**	t_R (*min*)	$t_R/t_{R,I}$ (-)	t_R-$t_{R,I}$ (*min*)	1^0 *m/z*	2^0 *m/z*	3^0 *m/z*	4^0 *m/z*
1)	1,4-DICHLOROBENZENE-D4	3.96	1.000	0.00	152	152		
2)	NAPHTHALENE-D8	5.21	1.000	0.00	136	68	108	
3)	Nitrobenzene-d5	4.52	0.868	-0.69	82	54	128	
4)	Naphthalene	5.23	1.004	0.02	128	127	129	
5)	2-Methylnaphthalene	5.88	1.129	0.67	141	141	115	
6)	ACENAPHTHALENE-D10	6.94	1.000	0.00	164	162	160	
7)	2-Fluorobiphenyl	6.23	0.898	-0.71	172	171	173	
8)	Acenaphthylene	6.78	0.977	-0.16	152	151	150	
9)	Acenaphthene **	6.97	1.004	0.03	154	153	76	
10)	Fluorene	7.49	1.079	0.55	166	165	167	
11)	PHENANTHRENE-D10	8.52	1.000	0.00	188	80	94	
12)	Phenanthrene	8.54	1.002	0.02	178	176	179	
13)	Anthracene	8.6	1.009	0.08	178	176	179	
14)	Fluoranthene	10.09	1.184	1.57	202	200	203	
15)	Terphenyl-D14	10.62	1.246	2.10	244	122	212	
16)	CHRYSENE-D12	12.5	1.000	0.00	240	236	120	
17)	Pyrene **	10.44	0.835	-2.06	202	101	200	
18)	Benzo(a)anthracene	12.46	0.997	-0.04	228	226	229	
19)	Chrysene	12.54	1.003	0.04	228	226	229	
20)	PERYLENE-D12	15.22	1.000	0.00	264	260	132	
21)	Benzo(b)fluoranthene	14.51	0.953	-0.71	252	253	126	
22)	Benzo(k)fluoranthene	14.56	0.957	-0.66	252	253	125	
23)	Benzo(a)pyrene	15.12	0.993	-0.10	252	253	126	
24)	Indeno(1,2,3-cd)pyrene	17.38	1.142	2.16	276	138	277	
25)	Dibenzo(ah)anthracene	17.39	1.143	2.17	278	139	279	
26)	Benzo(ghi)perylene	17.99	1.182	2.77	276	138	277	

†: Absolute retention times (t_R) listed are from calibration PF060829.M. Absolute and relative retention times ($t_R/t_{R,I}$) may shift with the present condition of the column (*i.e*. new, clipped, *etc*.), but the differential retention times (t_R - $t_{R,I}$) tend to remain constant given the same chromatographic temperature program.

Table 2a. Summary of Practical Quantitation Limits for Base/Neutral/Acid Semi-Volatile Organics †

COMPOUND	PQL_S (µg/kg)	PQL_W (µg/L)	IDL_S (µg/kg)	IDL_W (µg/L)	MDL_S (µg/kg)	MDL_W (µg/L)	PQL_S/IDL_S	PQL_W/IDL_W
Pyridine	330	10.0	18.6	0.559	24.9	0.8	17.9	17.9
N-Nitrosodimethylamine	330	10.0	12.7	0.380	32.4	0.6	26.3	26.3
2-Picoline	330	10.0	25.3	0.759	94.0	6.8	13.2	13.2
Methyl methanesulfonate	330	10.0	17.8	0.534	162.6	2.3	18.7	18.7
Ethyl methanesulfonate	330	10.0	9.0	0.271	150.1	3.8	36.8	36.8
Phenol **	330	10.0	14.6	0.439	278.0	4.0	22.8	22.8
Aniline	330	10.0	27.0	0.809	250.9	15.7	12.4	12.4
2-Chlorophenol **	330	10.0	14.8	0.443	261.4	3.6	22.6	22.6
Bis(2-chloroethyl)ether	330	10.0	7.9	0.238	247.3	4.4	42.0	42.0
1,3-Dichlorobenzene	330	10.0	15.9	0.476	226.3	5.1	21.0	21.0
1,4-Dichlorobenzene **	330	10.0	8.4	0.253	242.6	5.8	39.5	39.5
1,2-Dichlorobenzene	330	10.0	8.1	0.242	231.5	5.9	41.4	41.4
Bis(2-chloroisopropyl)ether	330	10.0	7.8	0.234	265.3	4.2	42.7	42.7
Benzyl Alcohol	330	10.0	10.9	0.328	307.0	4.5	30.5	30.5
Acetophenone	330	10.0	9.6	0.287	248.5	4.9	34.9	34.9
o-Cresol	330	10.0	8.3	0.249	257.4	3.3	40.1	40.1
p,m-Cresol	330	10.0	9.6	0.287	280.2	3.9	34.8	34.8
Hexachloroethane	330	10.0	13.0	0.390	222.5	4.3	25.7	25.7
N-Nitrosodi-n-propylamine*	330	10.0	11.2	0.336	305.9	5.5	29.8	29.8
Nitrobenzene	330	10.0	12.0	0.361	223.7	3.3	27.7	27.7
N-Nitrosopiperidine	330	10.0	15.7	0.471	243.3	4.0	21.2	21.2
Isophorone	330	10.0	10.1	0.304	238.6	4.0	32.9	32.9
2-Nitrophenol	330	10.0	59.8	1.793	208.3	4.0	5.6	5.6
2,4-Dimethylphenol	330	10.0	18.9	0.568	234.7	3.3	17.6	17.6
Bis(2-chloroethoxy)methane	330	10.0	13.2	0.397	232.1	4.3	25.2	25.2
2,4-Dichlorophenol	330	10.0	13.3	0.399	220.8	4.5	25.0	25.0
1,2,4-Trichlorobenzene **	330	10.0	16.5	0.494	209.0	4.1	20.2	20.2
Benzoic Acid	330	10.0	7.4	0.222	N/A	7.3	45.0	45.0
Naphthalene	330	10.0	12.7	0.380	222.2	4.9	26.3	26.3
4-Chloroaniline	330	10.0	118.1	3.542	307.7	9.2	2.8	2.8
2,6-Dichlorophenol	330	10.0	20.2	0.607	224.3	4.2	16.5	16.5
Hexachlorobutadiene	330	10.0	17.8	0.534	188.0	2.7	18.7	18.7
N-Nitroso-di-n-butylamine	330	10.0	17.8	0.535	256.1	4.6	18.7	18.7
4-Chloro-3-methylphenol **	330	10.0	12.6	0.377	264.8	4.2	26.5	26.5
2-Methylnaphthalene	330	10.0	14.4	0.433	223.4	4.5	23.1	23.1
1,2,4,5-Tetrachlorobenzene	330	10.0	22.2	0.667	197.6	3.5	15.0	15.0
Hexachlorocyclopentadiene	330	10.0	49.8	1.493	204.3	4.8	6.7	6.7
2,4,6-Trichlorophenol	330	10.0	16.1	0.483	212.0	3.2	20.7	20.7
2,4,5-Trichlorophenol	330	10.0	54.6	1.637	178.8	3.1	6.1	6.1
1-Chloronaphthalene	330	10.0	11.5	0.344	224.6	3.7	29.1	29.1
2-Chloronaphthalene	330	10.0	33.2	0.997	199.4	3.3	10.0	10.0
2-Nitroaniline	330	10.0	24.2	0.725	263.4	3.8	13.8	13.8
Acenaphthylene	330	10.0	40.4	1.212	247.1	3.4	8.3	8.3
Dimethyl Phthalate	330	10.0	10.2	0.305	322.0	3.8	32.8	32.8
2,6-Dinitrotoluene	330	10.0	24.4	0.732	315.2	3.9	13.7	13.7
3-Nitroaniline	330	10.0	27.8	0.833	368.1	5.1	12.0	12.0

COMPOUND	PQL_S (µg/kg)	PQL_W (µg/L)	IDL_S (µg/kg)	IDL_W (µg/L)	MDL_S (µg/kg)	MDL_W (µg/L)	PQL_S/IDL_S	PQL_W/IDL_W
Acenaphthene **	330	10.0	17.0	0.509	398.5	3.1	19.6	19.6
2,4-Dinitrophenol	330	10.0	63.4	1.902	N/A	5.7	5.3	5.3
Dibenzofuran	330	10.0	17.4	0.522	385.9	3.5	19.1	19.1
4-Nitrophenol **	330	10.0	145.4	4.361	249.7	8.0	2.3	2.3
2,4-Dinitrotoluene **	330	10.0	20.9	0.628	276.9	3.5	15.9	15.9
1-Naphthylamine	330	10.0	54.0	1.619	460.8	24.9	6.2	6.2
2-Naphthylamine	330	10.0	130.3	3.908	442.8	30.8	2.6	2.6
2,3,4,6-Tetrachlorophenol	330	10.0	17.4	0.521	245.5	2.9	19.2	19.2
Fluorene	330	10.0	13.2	0.396	348.9	3.8	25.2	25.2
Diethyl phthalate	330	10.0	17.2	0.516	298.5	3.8	19.4	19.4
4-Chlorophenyl phenyl ether	330	10.0	12.5	0.376	365.0	4.2	26.6	26.6
4-Nitroaniline	330	10.0	210.8	6.323	340.7	5.7	1.6	1.6
4,6-Dinitro-2-methylphenol	330	10.0	20.6	0.619	142.9	3.5	16.2	16.2
N-Nitrosodiphenylamine	330	10.0	14.8	0.445	290.2	4.5	22.5	22.5
1,2-Diphenylhydrazine	330	10.0	10.9	0.326	333.3	4.3	30.7	30.7
Azobenzene	330	10.0	10.9	0.326	333.1	4.3	30.7	30.7
4-Bromophenyl phenyl ether	330	10.0	9.0	0.270	314.3	3.9	37.0	37.0
1,3,5-Trinitrobenzene	330	10.0	19.9	0.596	246.9	4.8	16.8	16.8
Phenacetin	330	10.0	17.2	0.517	185.5	4.2	19.3	19.3
Hexachlorobenzene	330	10.0	13.3	0.399	279.8	4.3	25.1	25.1
4-Aminobiphenyl	330	10.0	96.7	2.900	495.4	29.9	3.4	3.4
Pentachlorophenol **	330	10.0	15.8	0.474	176.0	2.9	21.1	21.1
Pronamide	330	10.0	14.0	0.419	273.4	5.1	23.9	23.9
Pentachloronitrobenzene	330	10.0	24.6	0.738	282.6	4.9	13.6	13.6
Phenanthrene	330	10.0	9.3	0.280	263.3	4.0	35.8	35.8
Anthracene	330	10.0	16.7	0.500	245.5	4.3	20.0	20.0
di-N-butyl phthalate	330	10.0	13.1	0.394	201.5	5.1	25.4	25.4
Fluoranthene	330	10.0	15.0	0.450	225.2	4.5	22.2	22.2
Benzidine	330	10.0	78.9	2.368	59.1	25.4	4.2	4.2
Pyrene **	330	10.0	7.3	0.219	260.1	5.0	45.7	45.7
p-Dimethylaminoazobenzene	330	10.0	14.0	0.421	229.7	4.3	23.8	23.8
Butyl benzyl phthalate	330	10.0	5.0	0.151	361.7	4.8	66.0	66.0
Benzo(a)anthracene	330	10.0	7.5	0.226	269.4	5.6	44.3	44.3
3,3'-Dichlorobenzidine	330	10.0	15.0	0.450	326.5	16.8	22.2	22.2
Chrysene	330	10.0	8.0	0.240	273.4	5.4	41.6	41.6
Bis(2-ethylhexyl)phthalate	330	10.0	12.8	0.384	481.3	5.5	26.0	26.0
Di-n-octyl phthalate	330	10.0	20.8	0.625	330.7	5.0	16.0	16.0
Benzo(b)fluoranthene	330	10.0	21.5	0.646	267.5	6.7	15.5	15.5
7,12-Dimethylbenz(a)anthracene	330	10.0	19.0	0.571	230.3	6.8	17.5	17.5
Benzo(k)fluoranthene	330	10.0	17.7	0.530	269.6	5.6	18.9	18.9
Benzo(a)pyrene	330	10.0	22.5	0.675	253.1	5.2	14.8	14.8
3-Methylcholanthrene	330	10.0	12.3	0.369	264.6	4.9	27.1	27.1
Dibenz(a,j)acridine	330	10.0	17.3	0.520	49.4	6.0	19.2	19.2
Indeno(1,2,3-cd)pyrene	330	10.0	15.5	0.465	262.2	6.0	21.5	21.5
Dibenzo(a,h)anthracene	330	10.0	19.1	0.574	254.9	6.5	17.4	17.4
Benzo(ghi)perylene	330	10.0	8.8	0.263	273.9	5.7	38.1	38.1

†: Data are from 08/08/2001 (IDL data) and 02/04/2002 (MDL data).

Table 2b. Summary of Practical Quantitation Limits for Polynuclear Aromatic Semi-Volatile Organics†

COMPOUND	PQL_W (µg/L)	PQL_S (µg/kg)	IDL_W (µg/L)	IDL_S (µg/kg)	PQL_W/IDL_W	PQL_S/IDL_S
Naphthalene	5.0	330	0.957	31.9	5.2	10.4
2-Methylnaphthalene	5.0	330	0.478	15.9	10.5	20.9
Acenaphthylene	5.0	330	0.312	10.4	16.0	32.0
Acenaphthene	5.0	330	0.680	22.7	7.3	14.7
Fluorene	5.0	330	0.322	10.7	15.5	31.0
Phenanthrene	5.0	330	0.489	16.3	10.2	20.4
Anthracene	5.0	330	0.414	13.8	12.1	24.1
Fluoranthene	5.0	330	0.404	13.5	12.4	24.7
Pyrene	5.0	330	0.123	4.1	40.8	81.6
Benzo(a)anthracene	5.0	330	0.273	9.1	18.3	36.6
Chrysene	5.0	330	0.330	11.0	15.2	30.3
Benzo(b)fluoranthene	5.0	330	1.297	43.2	3.9	7.7
Benzo(k)fluoranthene	5.0	330	0.854	28.5	5.9	11.7
Benzo(a)pyrene	5.0	330	0.368	12.3	13.6	27.2
Indeno(1,2,3-cd)pyrene	5.0	330	0.300	10.0	16.7	33.4
Dibenzo(ah)anthracene	5.0	330	0.438	14.6	11.4	22.8
Benzo(ghi)perylene	5.0	330	0.624	20.8	8.0	16.0

†: Data are from 05/26/2001.

Table 2c. Practical Quantitation Limits for Diesel Range Organics†

COMPOUND	PQL_W (mg/L)	PQL_S (mg/kg)	IDL_W (mg/L)	IDL_S (mg/kg)	PQL_W/IDL_W	PQL_S/IDL_S
Diesel Range Organics	0.1	4	0.0078	0.26	15.5	12.9

†: Data are from 05/26/2001.

Table 3. GC/MS Operating Conditions - Base/Neutral/Acid (BNA) and Polynuclear Aromatic (PNA) Semi-Volatile Organics Analysis†

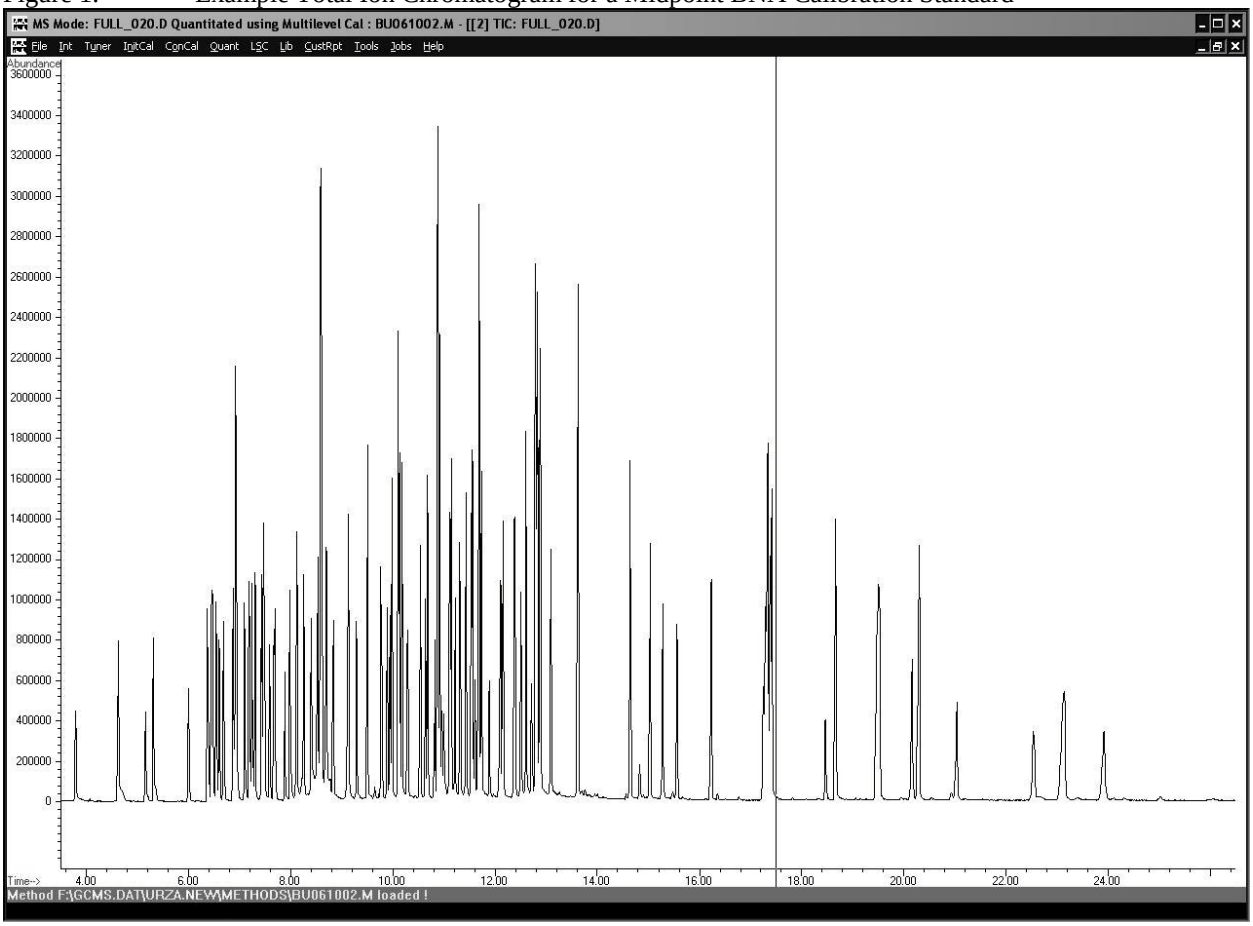
Operating Parameter	**BNA Analysis**	**PNA Analysis**
Chromatographic Column	HP-5MS SV, *L* = 30 *m*, *ID* = 0.25 *mm*	HP-5MS SV, *L* = 30 *m*, *ID* = 0.25 *mm*
Carrier Gas	Helium (He) at 2 *ml*/*min*	Helium (He) at 2 *ml*/*min*
Temperature Program	*Variable-Instrument dependent*	*Variable-Instrument dependent*
Injector Temperature	250°C	250°C
Detector Temperature	280°C	280°C
Injection Volume	1-2µ*l*	1µ*l*
Mass Scanning Range	40 *m*/*z* - 450 *m*/*z*	35 *m*/*z* - 550 *m*/*z*
Mass Scanning Rate	1.8 *Hz*	1.4 *Hz*

†: Note that DROs may be analyzed by either of the two sets of operating conditions.

Table 4. DFTPP Tune Evaluation Criteria

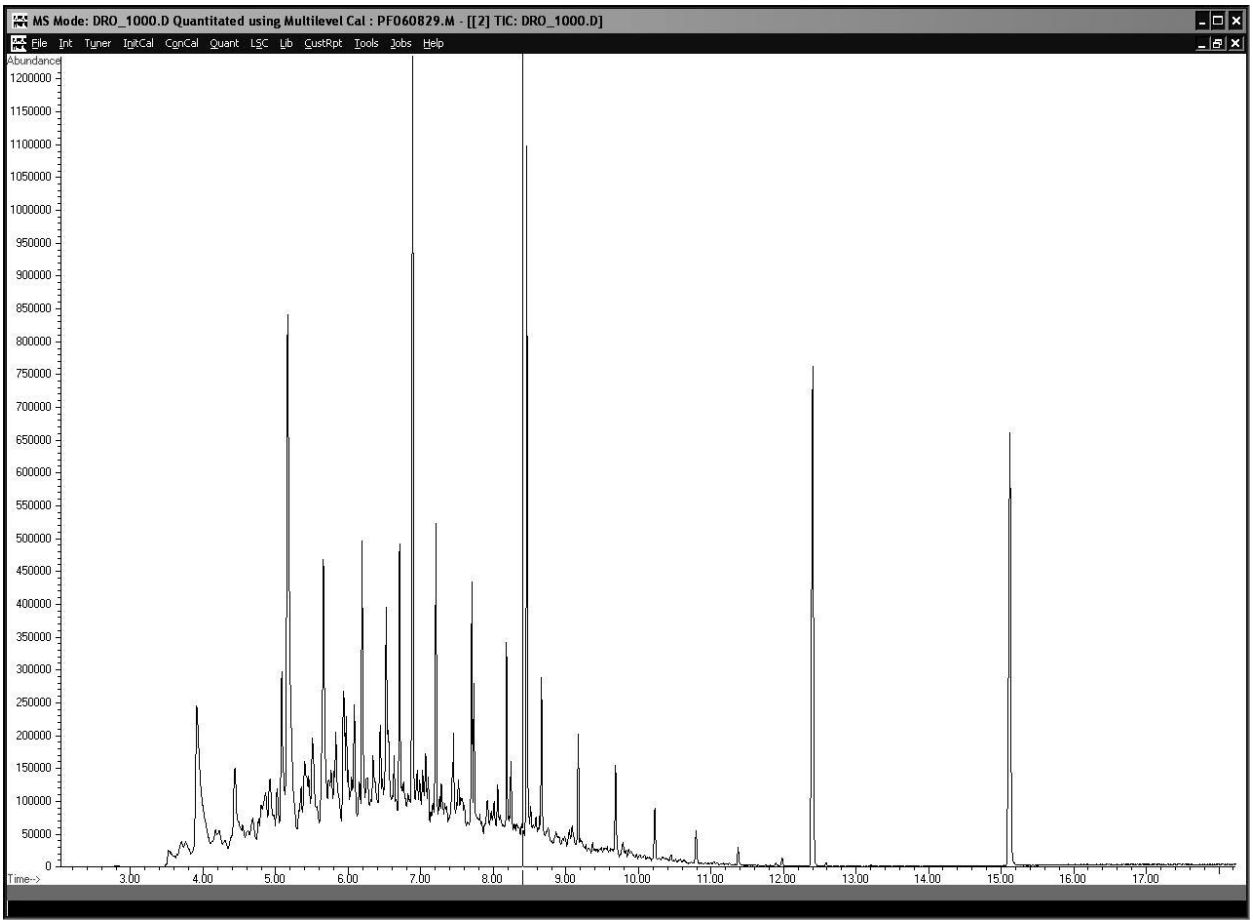
Target *m/z*	**Relative *m/z***	**LCL (%)**	**UCL (%)**
51	198	30.0	80.0
68	69	0.0	2.0
69	198	0.0	NA
70	69	0.0	2.0
127	198	25.0	75.0
197	198	0.0	1.0
198	198	100.0	100.0
199	198	5.0	9.0
275	198	10.0	30.0
365	198	1.0	NA
441	443	0.0	100.0
442	198	40.0	110.0
443	442	15.0	24.0

Figure 1. Example Total Ion Chromatogram for a Midpoint BNA Calibration Standard[†]



[†]: Data file FULL_020.D from calibration BU061002.M (20 ng/µL). GC/MS acquisition parameters are given by Table 4 for BNA Analysis.

Figure 2. Example Total Ion Chromatogram for a Diesel Range Organics Calibration Standard†



†: Data file DRO_1000.D from calibration PD061007.M (1.0 g/L). GC/MS acquisition parameters are given by Table 4.